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CREEP OF COPPER

AT

INTERMEDIATE TEMPERATURES

THIRTY-EIGHTH TECHNICAL REPORT

BY

T. E. TIETZ
J. E. DORN

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MINERALS RESEARCH LABORATORY
INSTITUTE OF ENGINEERING RESEARCH
UNIVERSITY OF CALIFORNIA, BERKELEY

September 24, 1954
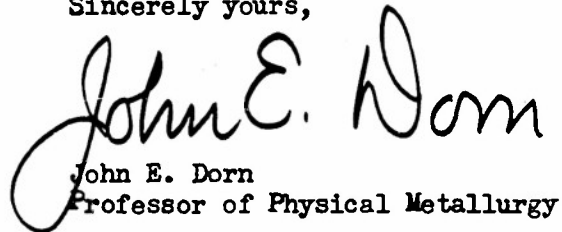
Office of Naval Research
Department of the Navy
Washington 25, D. C.

ATTENTION: Mr. Julius Harwood

Dear Sir:

Attached hereto is a copy of the Thirty Eighth Technical
Report on Contract N7-onr-295, Task Order II, entitled "Creep
of Copper at Intermediate Temperatures" which was based on a
thesis submitted by Mr. T. E. Tietz in partial satisfaction of
the requirements for the Ph.D. degree in Physical Metallurgy.
The context of this report reveals that creep at intermediate
temperatures obeys essentially the same laws as apply to high
temperature creep with the singular exception that the activa-
tion energy for creep in the intermediate temperature range is
less than that for self-diffusion.

Sincerely yours,

John E. Dorn
Professor of Physical Metallurgy

JED:dk

CREEP OF COPPER

AT

INTERMEDIATE TEMPERATURES

By

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ABSTRACT

Activation energies for creep of copper at intermediate temperatures, where crystal recovery was negligible, were determined by the simple technique of rapidly alternating the test temperature between T_1 and T_2 ($T_2 = T_1 +$ about $10°K$) throughout a constant stress creep test. The activation energy for a given state and stress was determined from the well-known relationship

$$\dot{\varepsilon}_1 \, e^{\frac{\Delta H}{RT_1}} \;=\; \dot{\varepsilon}_2 \, e^{\frac{\Delta H}{RT_2}}$$

where $\dot{\varepsilon}$ = creep rate

e = base of natural logarithms

R = gas constant

T = absolute temperature

ΔH = energy supplied by thermal activation

and the subscripts refer to the conditions immediately preceding and immediately following a change in temperature. ΔH was found to be 37,000 $\pm$3,000 cal/mole independent of stress and strain.

The same creep laws as have been previously established for high temperature creep were found to be valid for creep at intermediate temperatures. But the ΔH was found to be lower than that for self-diffusion in the intermediate temperature range whereas it is known to be equal to that for self-diffusion at high temperatures.

INTRODUCTION

It is now well established that creep at high temperatures can be correlated by the functional relationship[1-4]

$$\varepsilon = f(\theta), \qquad\qquad \sigma = \text{const.} \qquad (1)$$

where ε = total plastic strain,

f = function that depends on the stress,

$$\theta = \int_0^t e^{-\frac{\Delta H}{RT}} dt,$$

t = duration of test,

e = base of natural logarithms,

ΔH = activation energy for creep,

R = gas constant,

T = absolute temperature,

and σ = stress.

Thus identical values of θ are achieved during creep under a given stress at two alternate temperatures at the same total strain. At the same strain therefore

$$t_1 e^{-\frac{\Delta H}{RT_1}} = t_2 e^{-\frac{\Delta H}{RT_2}} \qquad (2)$$

where the subscripts refer to the two alternate temperature conditions of test. Consequently the activation energy for high temperature creep is readily obtained with the aid of Eq. 2. Activation energies so obtained have been observed to be insensitive to the creep strain at which they are evaluated, the applied stress, grain size, minor alloying additions,[1-4] cold worked state[5], and the presence of thermally stable dispersions of intermetallic compounds in an alpha solid solution matrix.[6] Furthermore the activation energy for high temperature creep is very nearly equal to

that for self-diffusion in all cases where adequate data are available for comparison.[3,7]

The apparent identity of the activation energies for high temperature creep and self-diffusion strongly suggest that high temperature creep arises from a dislocation climb process. This hypothesis is further strengthened by the fact that the energy of activation for high temperature creep is insensitive to the applied stress[18], and by the observations that the correlation suggested by Eq. 1 cannot be extended to temperatures below those for rapid crystal recovery.[1]

When using the activation energy for self-diffusion, Eq. 1 fails to give the required correlations between creep curves at various intermediate temperatures.[1] This suggests that the dislocation climb mechanism for creep might be superseded by some alternate mechanism in the intermediate range of temperatures. This investigation was initiated in a preliminary attempt to ascertain the type of creep law that might be valid in the intermediate range of temperatures. Special consideration, however, was given to the determination of the activation energy for creep in this range because the Becker-Orowan[8,9] and the Mott-Nabarro[10] theories for transient creep suggest that the activation energy should increase with strain and the Kauzmann-Seitz[11,12] types of thermal activation theories suggest further that the activation energy should decrease with increasing values of the applied stress. Thus a knowledge of the effect of strain or stress on the activation energy for creep might provide definitive information on several possible mechanisms for creep.

EXPERIMENTAL TECHNIQUE

The creep rate continually changes during isothermal-constant stress creep tests. Since all of the externally controllable variables are maintained constant, the change in the creep rate must be ascribed to structural

changes that attend the process of creep. For example, it has been shown that the success of Eq. 1 for high temperature creep is attributable in part to the fact that identical structures are produced at the same values of Θ for creep at a given stress, independent of the test temperature.[2] Special precautions must therefore be taken in any evaluation of the activation energy for creep, so that the value obtained is a true activation energy that is free from possible errors arising from neglect of structural differences.

A simple and direct technique for determining the activation energy for creep was used in the present investigation. As shown by the typical example given in Fig. 1 this technique consisted of alternating the temperature between T_1 and T_2 throughout a constant stress creep test. Since no abrupt changes in strain occurred and no spurious transients were detected in the creep rates, the structure was presumed to be the same the instant before and the instant following the rapid change in temperature. Consequently, for each point of temperature change, a true activation energy for creep at the given stress could be obtained from the well-known relationship

$$\dot{\varepsilon}_1 e^{\frac{\Delta H}{R T_1}} = \dot{\varepsilon}_2 e^{\frac{\Delta H}{R T_2}} \tag{3}$$

where $\dot{\varepsilon}$ is the creep rate and the subscripts refer to the conditions just preceding and immediately following an abrupt change in temperature. This technique involves only the assumption that creep occurs by means of some thermal activation process; it is valid for any creep mechanisms that depend on thermal activation. Only one specimen is used in evaluating the activation energy and therefore the effects of sampling scatter on the calculated activation energy are eliminated; the accuracy of the activation
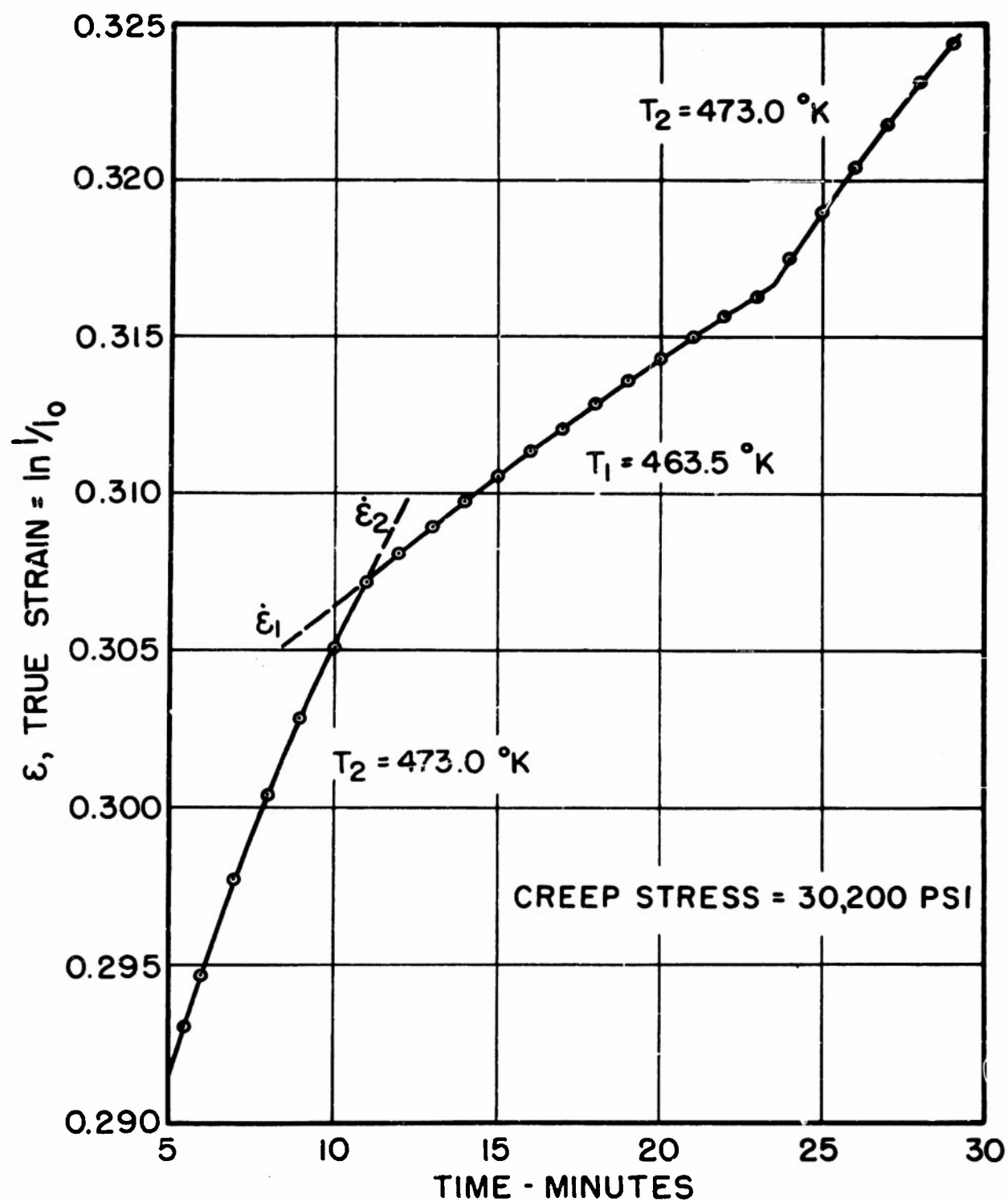
FIG. 1   CREEP CURVE SHOWING TWO
TEMPERATURE CHANGES.

energy so obtained depends exclusively on the accuracy with which the strain rates and temperatures can be determined.

A technique similar to that described here has been applied by Kuhlmann and Masing[13] and Lucas and Masing[14] to investigations on the creep of hard drawn springs of aluminum and copper.

TEST EQUIPMENT

The experimental equipment that was used in this investigation is identified in Fig. 2. The load was applied to the specimen by means of a special Andrade[15] type contoured lever arm that was designed so as to maintain a constant stress on the specimen throughout a creep test. As the specimen elongated its cross-sectional area decreased and the lever arm correspondingly decreased to maintain the stress constant. Small deviations in strain between the gage section and the total reduced section, however, led to minor variations in the true stress on the specimen. In order to ascertain the variations in stress a proving ring was placed in series with the specimen. The maximum variation in stress did not exceed about 1.2% throughout any one test and was usually much less than this amount.

Special silicone oil temperature baths were employed in order to obtain necessary accuracy in the test temperatures. Thermocouples on the specimen showed that the steady state temperature did not vary more than about $\pm 0.1°C$ throughout a test. Upon change in temperature less than 4 minutes were required for the specimen to reach $\pm 0.2°C$ from the steady state value. The creep strain rates were evaluated only after the steady state temperature was reached and, as shown in the context, they were extrapolated through the transient temperature range to the instantaneous value following the instant the temperature was changed.

CONSTANT STRESS
CONTOUR ARM

PROVING RING

EXTENSOMETER
DIAL

APPLIED LOAD

BATH AT T_2

BATH AT T_1

FIG. 2 CREEP TESTING MACHINE.

As shown in Fig. 3 the creep strains were measured by means of a concentric rod and tube type extensometer which actuated a 0.0001 in. dial gage.

TEST MATERIAL

Several simple requirements were demanded of the metal to be used in this investigation:

1. The metal should not exhibit rapid crystal recovery at the highest usable temperatures of the silicone oil baths, namely about $470°K$.

2. It should be relatively pure in order to provide reasonable freedom from possible auxiliary solid solution effects.

3. The metal should exhibit accurately determinable creep rates in the range from about $300°K$ to $475°K$ over a reasonably broad range of stresses and strains.

A few preliminary investigations demonstrated that OFHC copper exhibited the required specifications. Tensile creep specimens of OFHC copper were machined from 0.125 in. thick sheet material with their axes parallel to the rolling direction. Each specimen was 0.250 in. wide and had a reduced section of 1.500 in., the central 1.000 inch of which constituted the gage section. After machining, the specimens were annealed in a helium atmosphere for $1\frac{1}{2}$ hours at $748°K$ and then furnace cooled. The resulting average grain diameter was 0.02 mm.

A check on the recovery of the OFHC copper is illustrated in Fig. 4. A specimen was allowed to creep to about 11% total strain during which period of time the creep rate decreased several orders of magnitude due to the structural changes induced by the creep deformation. At this point the stress was removed in order to permit recovery at the test temperature
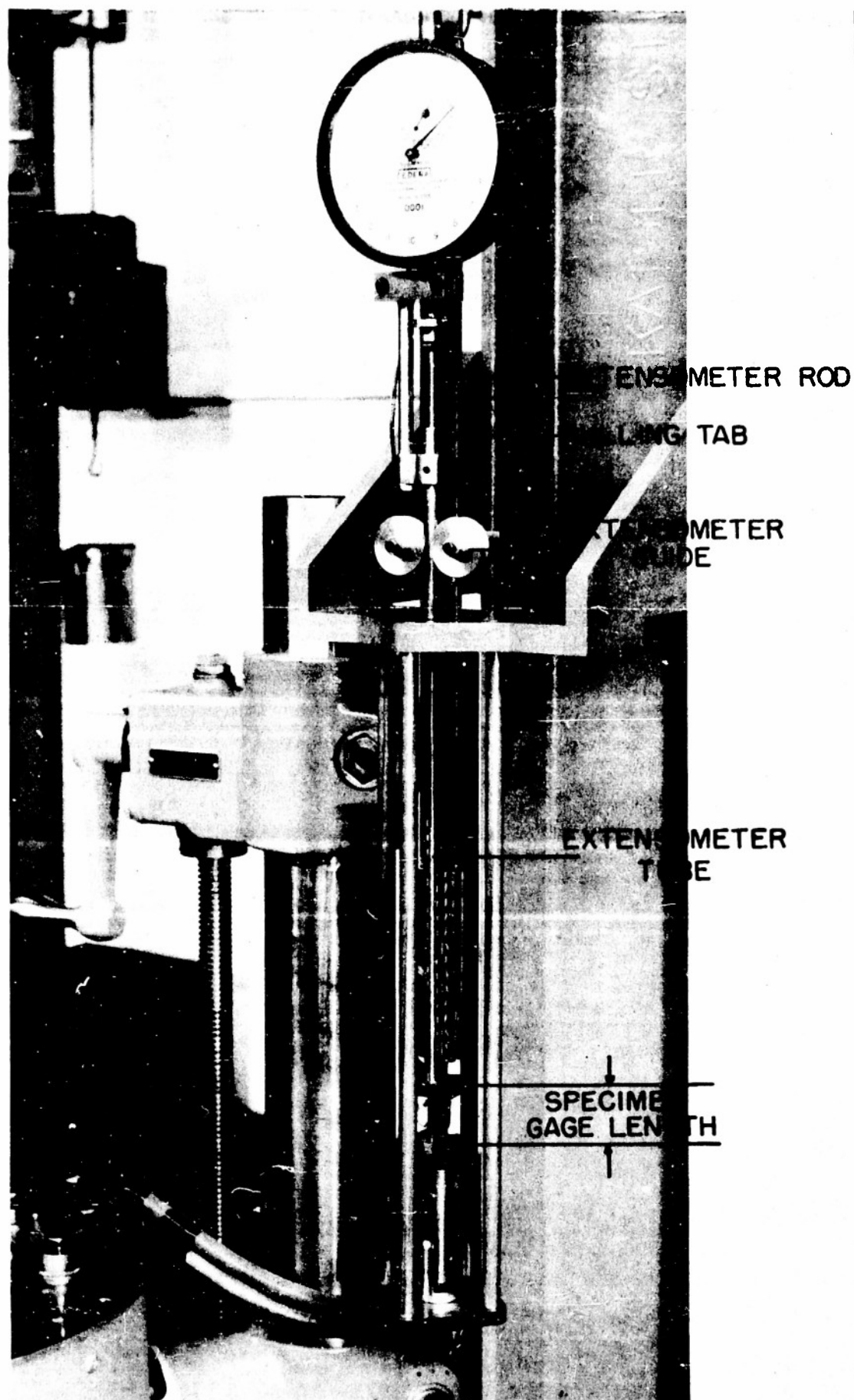
FIG. 3 CREEP SPECIMEN AND EXTENSOMETER
IN TEST POSITION.

of 473.5°K. Following a period of one hour the stress of 19,200 psi was reapplied. The fact that the creep curve continued as if the test had not been interrupted for annealing suggests that recovery under zero stress of the structural changes induced by the preceding creep was indeed negligible.

EXPERIMENTAL RESULTS

Creep rates were determined by graphical differentiation of the original creep curves such as that illustrated in Fig. 1. Such derived creep rate versus total plastic strain curves are given in Figs. 5 to 11. T_M is the mean temperature, ε_o the initial strain on loading and $\overline{\Delta H}$ the average activation energy of the test. The instantaneous activation energy for each change in temperature was determined from the two test temperatures and the extrapolated strain rates at the instant the temperature was changed in accord with Eq. 3.

DISCUSSION

A detailed summary of all activation energies that were determined is recorded in Fig. 12. These data reveal that the activation energy for creep of OFHC copper over the range of intermediate temperatures from 348°K to 469°K is about 37,000 ±3,000 calories per mole, independent of the total strain from $\varepsilon \cong 0.03$ to 0.38, and independent of the applied stress from $\sigma \cong 9,900$ to 38,300 psi. Obviously neither the Becker-Orowan and Mott-Nabarro nor the Kauzmann-Seitz types of creep theories apply to creep in the intermediate range of temperatures. The insensitivity of the activation energy to stress or strain is somewhat reminiscent of high temperature creep. However the activation energy for high temperature creep is expected to be that for self-diffusion, namely about 48,000 calories per mole for

FIG. 5 CREEP RATE – STRAIN CURVES FOR USE IN
THE EVALUATION OF ΔH FOR OFHC COPPER.

FIG. 6 CREEP RATE – STRAIN CURVES FOR USE IN THE
EVALUATION OF ΔH FOR OFHC COPPER.

FIG. 7 CREEP RATE – STRAIN CURVES FOR USE IN
THE EVALUATION OF ΔH FOR OFHC COPPER.

FIG. 8 CREEP RATE – STRAIN CURVES FOR USE IN
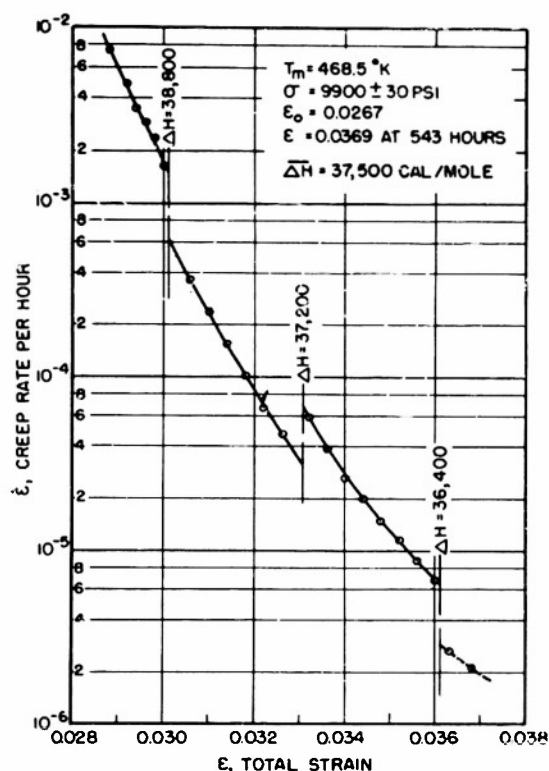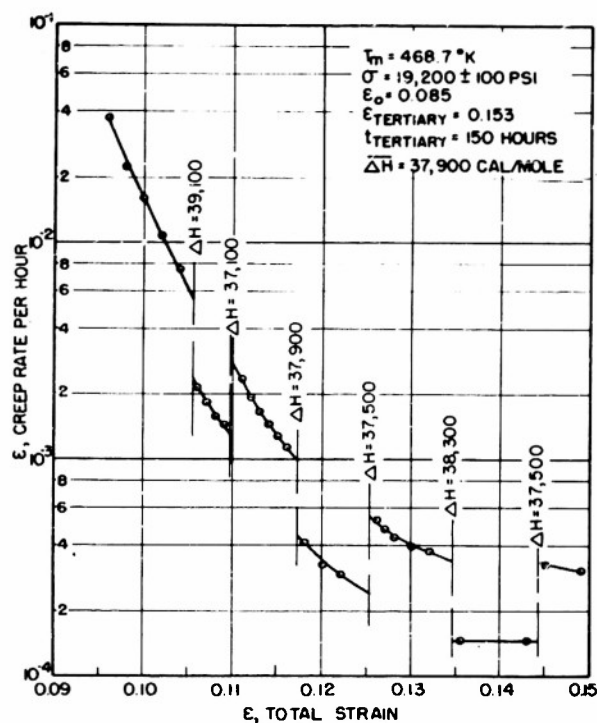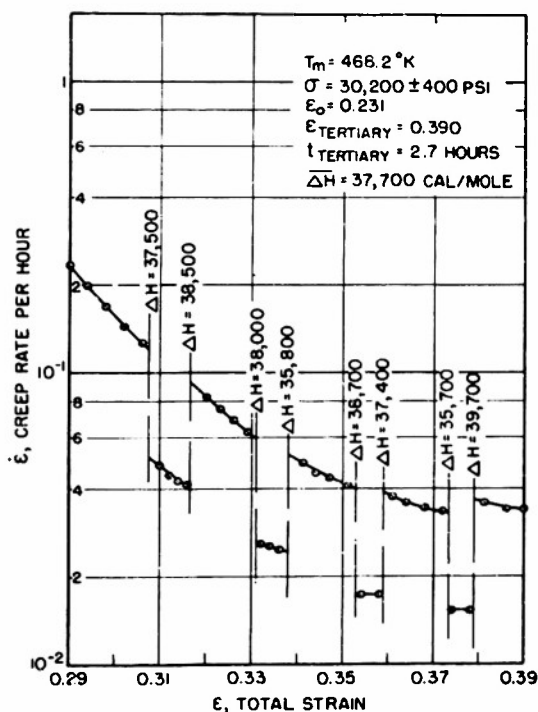THE EVALUATION OF ΔH FOR OFHC COPPER.
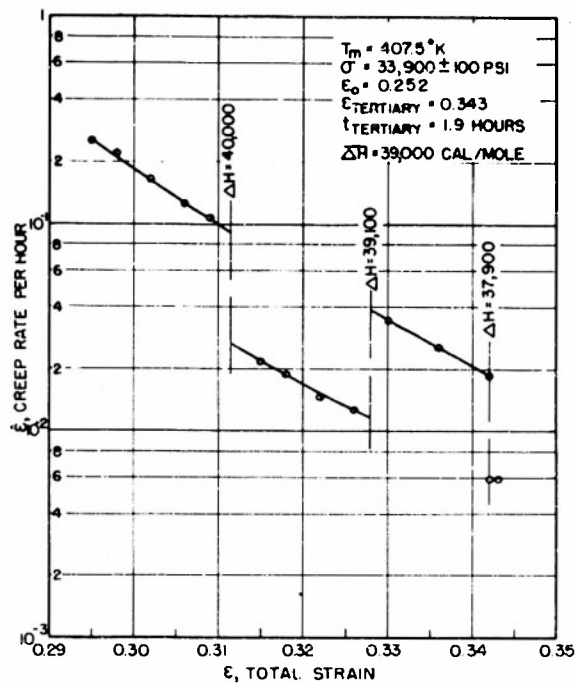
FIG. 9 CREEP RATE-STRAIN CURVES FOR USE IN THE
EVALUATION OF ΔH FOR OFHC COPPER.
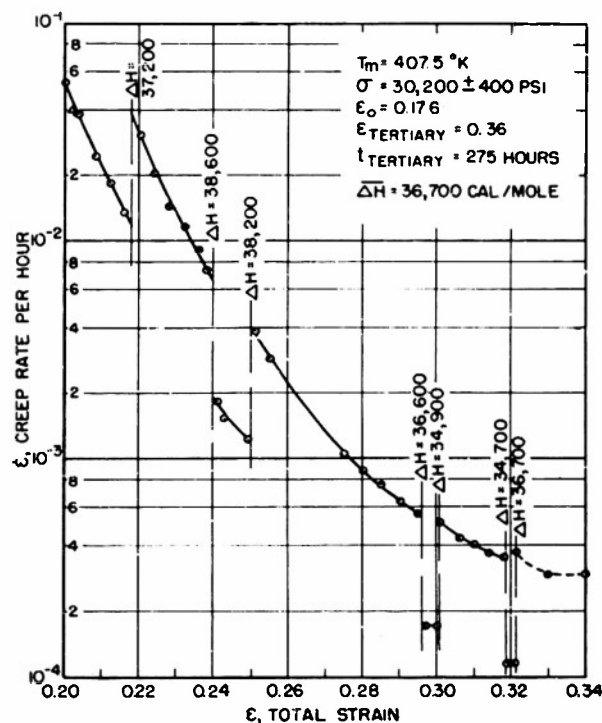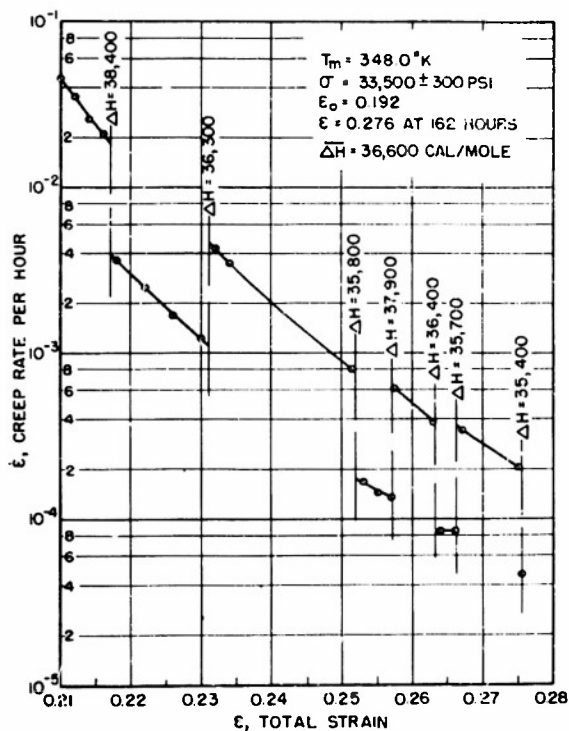

FIG.10 CREEP RATE-STRAIN CURVES FOR USE IN
THE EVALUATION OF ΔH FOR OFHC COPPER
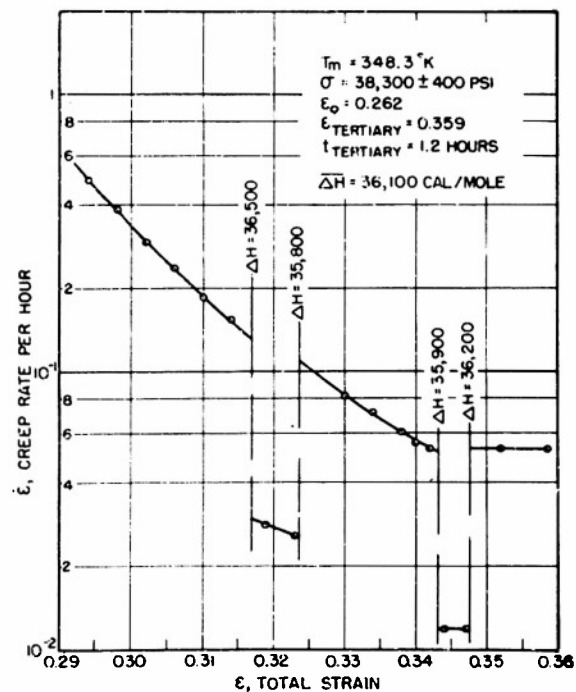
FIG. 11 CREEP RATE-STRAIN CURVES FOR USE IN THE
EVALUATION OF ΔH FOR OFHC COPPER.

SYMBOL		T_m, °K	σ, PSI	$\overline{\Delta H}$ CAL/MOLE
●, ○		468.5	9,900	37,500
■, □		468.7	19,200	37,900
◆, ◇		468.2	30,200	37,700
▲, △		407.5	30,200	36,700
▶, ▷		407.5	33,900	39,000
▣, ▫		348.0	33,500	36,600
▣, ▫		348.3	38,300	36,100

TEMPERATURE CHANGE

SOLID SYMBOL $T_2 \longrightarrow T_1$

OPEN SYMBOL $T_1 \longrightarrow T_2$

WHERE $T_2 \cong T_1 + 10$ °K

FIG. 12 ENERGY OF ACTIVATION AS A FUNCTION OF STRAIN.

copper.[3] The value obtained here, namely 37,000 calories per mole, suggests that a somewhat different mechanism for creep applies over the intermediate range of temperatures.

Additional insight into the details of creep at intermediate temperatures can be gained by further analysis of the data reported here. The decreasing creep rates obtained during the course of isothermal creep at constant stress are ascribable to changes in structure. Since the ΔH of the present investigation is insensitive to the strain it is insensitive to the structural changes attending creep. Consequently, in the intermediate as well as the high temperature range of creep,
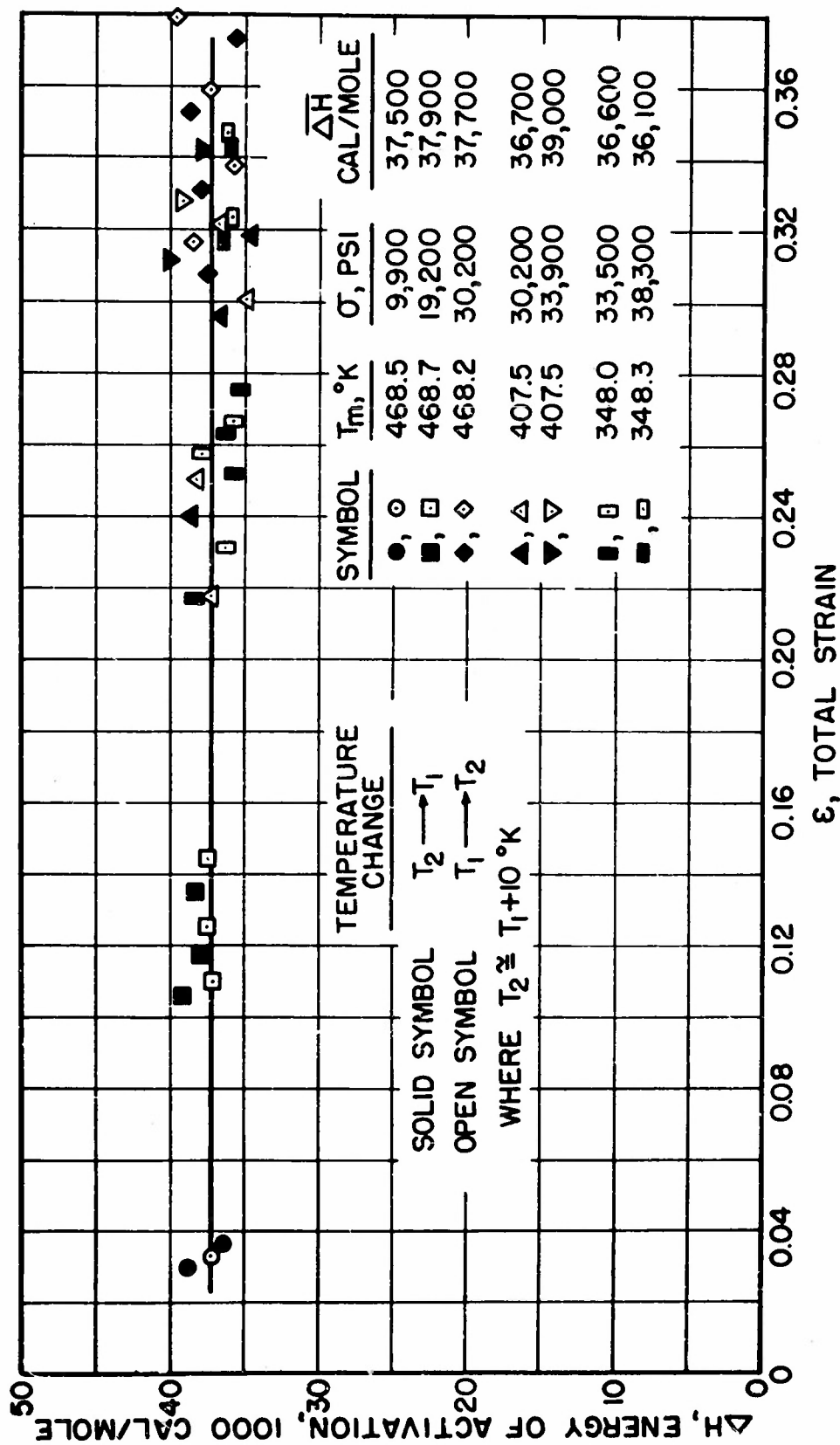
$$\dot{\varepsilon}\, e^{\frac{\Delta H}{RT}} = f\,(\text{Structure}), \qquad\qquad \sigma = \text{const.} \qquad (4)$$

Previous investigations have shown that the structure obtained for high temperature creep at a given stress depends only on the total strain, ε , or on Θ , independent of the test temperature. The question naturally arises as to whether this law is also valid over the intermediate temperature range or whether the structure depends on other types of relationships.

In Fig. 13, $\dot{\varepsilon}\, e^{\frac{\Delta H}{RT}}$ for a stress of 30,200 psi is plotted as a function of the total strain, ε , for each of two mean temperatures of 407.5° and 468.2°K. Similarly in Fig. 14, $\dot{\varepsilon}\, e^{\frac{\Delta H}{RT}}$ is plotted for almost identical stresses of about 33,700 psi as a function of the total strain, ε , for each of the two mean temperatures of 348.0° and 407.5°K. Both sets of data reveal that

$$\dot{\varepsilon}\, e^{\frac{\Delta H}{RT}} = F\,(\varepsilon), \qquad\qquad \sigma = \text{const.} \qquad (5)$$

Consequently the structure obtained during creep of an annealed specimen at a given stress in the intermediate range of temperatures depends on
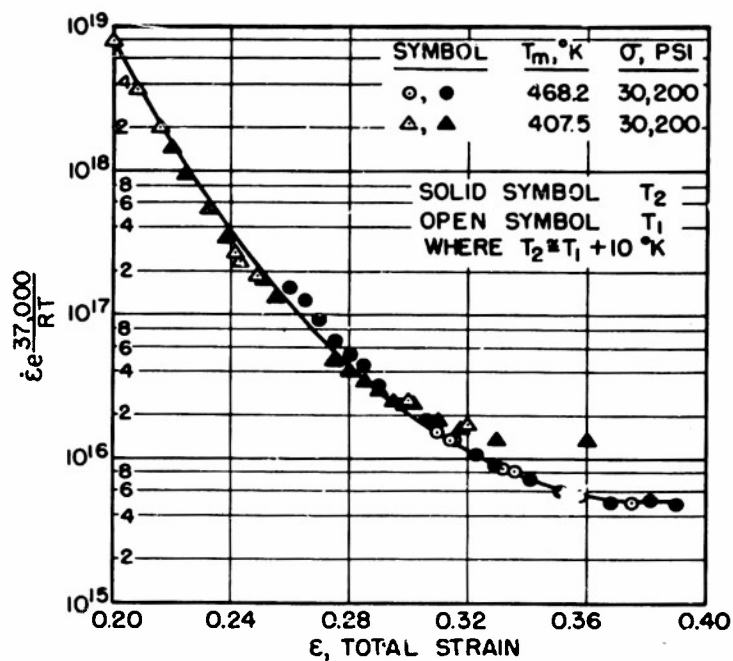
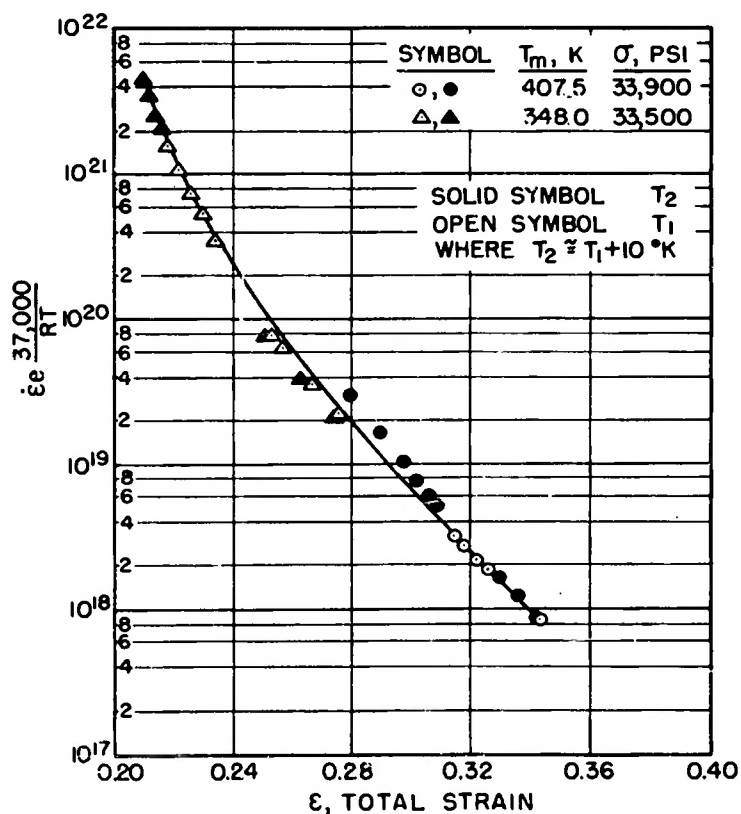FIG. 13 $\dot{\varepsilon}e^{\frac{\Delta H}{RT}}$ AS A FUNCTION OF ε.

FIG. 14 $\dot{\varepsilon}e^{\frac{\Delta H}{RT}}$ AS A FUNCTION OF ε.

the strain and is independent of the test temperature in harmony with the corresponding high temperature creep law.

Inasmuch as recovery of the structural changes induced by creep of annealed copper was found to be negligible in the intermediate range of temperatures under investigation here, and inasmuch as the temperature differences were reasonably modest, the above conclusion might have been anticipated. On the other hand, the absence of recovery and the differences in the initial strain for creep at two alternate temperatures might suggest that the structure developed during creep at a given stress in the intermediate range of temperatures might not be a unique function of Θ . However as shown by the data given in Figs. 15 and 16, this conclusion appears to be incorrect, and

$$\dot{\varepsilon}\, e^{\frac{\Delta H}{RT}} = \frac{d\varepsilon}{d\Theta} = \psi(\Theta) , \qquad \sigma = \text{const.} \qquad (6)$$

for both high and intermediate temperature creep. This is illustrated more forcefully by integrating Eq. 6 with respect to Θ which gives the well-established Eq. 1 for high temperature creep. Thus both high and intermediate temperature creep obey the same laws as demanded by Eqs. 1, 5, and 6. The difference between creep in these two ranges arises from the fact that the activation energy for high temperature creep is that for self-diffusion whereas that for creep at intermediate temperatures is significantly less than that for self-diffusion. It is this difference rather than a failure of the laws of high temperature creep that was responsible for the poor correlations obtained by means of Eq. 1 in the intermediate range of temperatures when the ΔH for self-diffusion was applied.

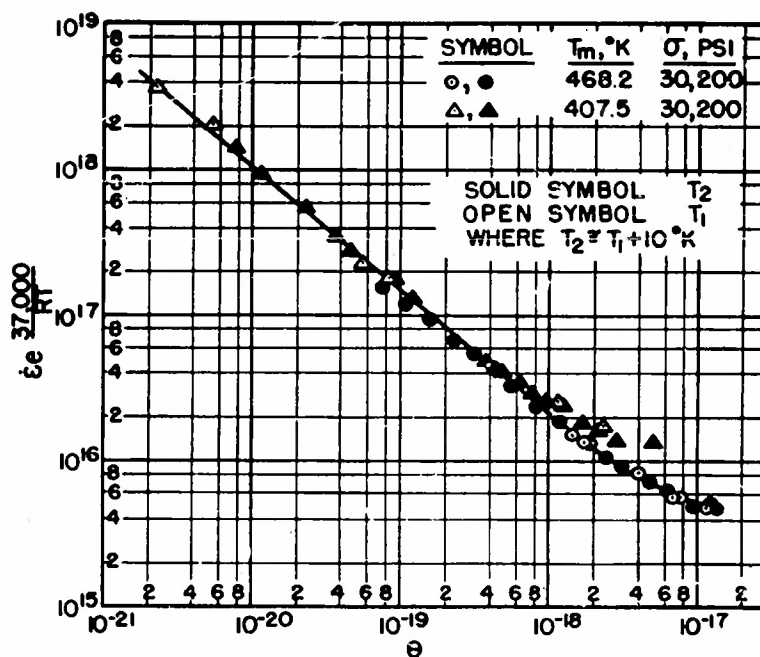Whereas theoretical support can be given for the coincidence of the

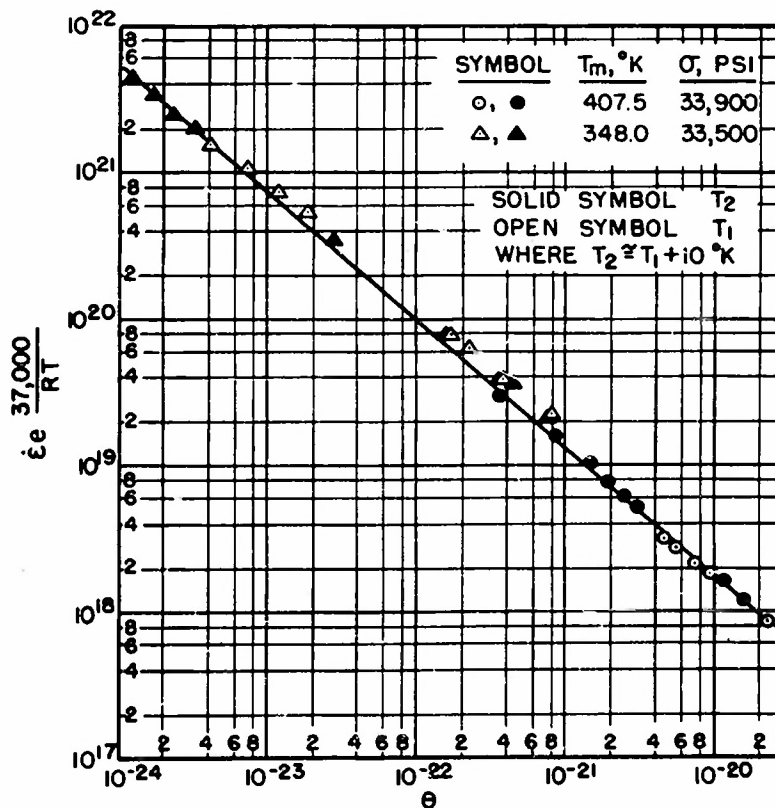FIG. 15 $\dot{\epsilon}e^{\frac{\Delta H}{RT}}$ AS A FUNCTION OF θ.

FIG. 16 $\dot{\epsilon}e^{\frac{\Delta H}{RT}}$ AS A FUNCTION OF θ.

activation energy for high temperature creep and that of self-diffusion, the authors have not yet been able to justify the activation energy of 37,000 calories per mole for the creep of copper in the intermediate temperature range. A simple dislocation climb model for high temperature creep suggests that as edge dislocations that are impeded by barriers climb out of their slip planes, the back stress that they induce on their Frank-Read sources decreases and new dislocations are released. Consequently the creep rate should be proportional to the rate of dislocation climb which is dependent on the product of the number of vacancies present per unit volume, n_v , and the probability of an atom-vacancy exchange. Therefore

$$\dot{\varepsilon} \propto n_v e^{-\frac{E_e}{RT}}$$

where E_e is the activation energy for an atom-vacancy exchange. Seitz has suggested that the number of vacancies produced during creep is proportional to the creep strain.[16] But at high temperatures such vacancies should diffuse rapidly to various types of sinks so that approximately the equilibrium number of vacancies might be present.

Under these conditions

$$n_v \propto e^{-\frac{E_v}{RT}}$$

where E_v is the energy necessary to produce a vacancy. Consequently the activation energy for high temperature creep would be ($E_e + E_v$) which is that for self-diffusion. The similarity of the high and intermediate temperature laws for creep suggests that creep at intermediate temperatures might also arise from a dislocation climb process. At such intermediate temperatures, however, the number of vacancies per unit volume might well

exceed the equilibrium number. Thus the activation energy for creep at intermediate temperatures might approximate E_e. However, according to Brinkman et al.[17] the activation energy for atom-vacancy exchange as determined by recovery of the electrical resistivity of cold worked copper is about 28,000 calories per mole which is appreciably less than the value of 37,000 calories per mole obtained for creep at intermediate temperatures. Furthermore some dislocation climb and partial recovery should have occurred during the interrupted creep test previously described if a dislocation climb model were valid in the intermediate temperature range. It therefore appears that additional investigations will be required in order to elucidate more completely the mechanism of creep at intermediate temperatures.

CONCLUSIONS

1. The activation energy for creep of copper over the intermediate range of temperatures from $348°K$ to $469°K$ was found to be $37,000 \pm 3,000$ calories per mole independent of the applied stress and the creep strain.

2. Neither the Becker-Orowan, Mott-Nabarro nor the Kauzmann-Seitz types of creep mechanisms agree with the experimental results on creep at intermediate temperatures.

3. Creep at intermediate temperatures obeys the same general laws as creep at high temperatures, namely

$$\dot{\varepsilon} \, e^{\frac{\Delta H}{RT}} = F(\varepsilon) = \psi(\Theta), \qquad \sigma = \text{const.}$$

But the activation energy for creep at intermediate temperatures differs from that for high temperature creep in that it is less than that for self-diffusion.

4. Additional investigations will be required in order to elucidate the mechanism of creep at intermediate temperatures.

ACKNOWLEDGMENTS

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